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NAVORD REPORT 6632

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THE ELECTROSTATIC SPARK SENSITIVITY OF BULK EXPLOSIVES AND METAL/OXIDANT MIXTURES (U)

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U. S. NAVAL ORDNANCE LABORATORY
WHITE OAK, MARYLAND

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NAVWEPS Report 6632

THE ELECTROSTATIC SPARK SENSITIVITY OF BULK EXPLOSIVES AND METAL/OXIDANT MIXTURES(U)

Prepared by:
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Approved by: ________________
Chief, Explosion Dynamics Division

ABSTRACT: In order to have a better basis for comparing explosive electro-static sensitivity measurements made in the United Kingdom (Explosives Research & Development Establishment, Waltham Abbey), and the United States (Naval Ordnance Laboratory, White Oak, Maryland) a replica of the E.R.D.E. apparatus has been built and used at NOL. Substantially identical results on similar explosives were obtained on the original E.R.D.E. apparatus and on its replica. A number of explosives were tested for the first time on the E.R.D.E. type of apparatus: namely metal/oxidant mixtures containing zirconium, boron, or titanium, graphite coated ammonium perchlorate, ammonium perchlorate/aluminum, GR propellant, HMX, and DATB. Other runs were made to compare the relative sensitivities of primary and secondary explosives and, where possible, relate these results to those obtained in the U.K.

By the work performed it was found that there is no large variation in sensitivity of normal lead styphnate with particle size; the sensitivity of explosives and reactive mixtures is somewhat affected by the polarity of the electrodes; the sensitivity of metal-oxidant mixtures based on zirconium, boron, or titanium is about that of primary explosives; and ammonium perchlorate mixtures are insensitive to electrostatic discharges.

* At Explosives Research and Development Establishment, Waltham Abbey, England
This work was carried out while Dr. Wyatt was in residence at NOL as a visiting research scientist.

Explosions Research Department
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Dr. R. M. H. Wyatt of the E.R.D.E., Waltham Abbey, United Kingdom, was assigned to the NOL for a period of two years as an exchange research scientist. While at NOL he pursued, along with other explosive sensitivity problems, the electrostatic sensitivity of explosives, work he was originally associated with in the U. K. The work was done to develop better methods for obtaining comparable results between NOL and U. K. tests and to study certain new explosive materials. The present report is a resume of Dr. Wyatt's work in the electro-static sensitivity field during his tour of duty at NOL. It should be of interest to scientists working the field of explosive sensitivity and the process of initiation.

This work was carried out under Task No. 507-525/53022/01040, Guided Missile Fuze Explosive Train Research. These studies bear on Explosives Research Key Problem 7.7.7 — Investigate the basic mechanism of initiation of explosives; develop new and more reliable tests for sensitivity — listed in NavOrd Report 3906.

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Commander

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By direction

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THE ELECTROSTATIC SPARK SENSITIVITY OF BULK EXPLOSIVES AND METAL/OXIDANT MIXTURES

INTRODUCTION

The Original NOL Spark Sensitivity Equipment

1. The spark sensitivity tester at NOL (described in NOLM 9959, reference (a), and subsequently modified) employs a variable vacuum capacitor of range 0-500 micro-microfarads (mmfd) and a DC power supply of range 0-20 KV. The approach limit of the electrode gap is set at 0.03 inch, the potential at 5,000 volts, and the capacity varied to alter the energy of the spark. For an actual test the spring loaded upper needle electrode is brought rapidly down toward the fixed base electrode (upon which the explosive is placed) by giving it a sharp blow with the hand.

The Spark Sensitivity Work at E.R.D.E.

2. The author and his colleagues at the Explosives Research and Development Establishment (E.R.D.E.), Waltham Abbey, England, have carried out experiments on the electrostatic spark sensitivity of bulk explosives over a wide range of capacity and voltage with an apparatus similar to, but not identical with, that discussed above. The principle difference between the two equipments is that the one in the U. K. is such that the upper electrode* can be made to make contact, if necessary, with the base electrode. Moreover, the base electrode can be either steel or a non-metal, the latter simulating more closely an accidental static discharge involving an operator.

* In this report the electrodes will be designated by the combination of materials used with the upper electrode always given first. For example, steel/rubber signifies that the upper electrode was steel and the base electrode rubber. Steel/steel would signify a steel upper electrode and a steel base electrode.

3. The following conclusions can be drawn from the work in the U. K. described in references (b), (c), (d), (e), and (f).

(a) Using metal/metal electrodes there are two regions of ignition of lead and silver azides - a low energy region involving short arc discharges and a high energy region involving gaseous discharges.

(b) For lead styphnate and LDNR* there is not a sharp distinction between the two regions mentioned in (a) above. This is evidenced by the fact that there is little discontinuity in the curve of percentage ignition with energy. (These two explosives are considerably more sensitive to gaseous discharges than the two azides.)

(c) Lead azide (i.e. of 97 percent or more purity) is more sensitive than lead styphnate to electrical discharges between metal/metal electrodes if both regions of ignition are taken into account. This is opposite to what is experienced in practice when manufacturing the two explosives and filling them into detonators, primers, etc. Many accidents with lead styphnate have been caused through ignition by stray electrical discharges; whereas lead azide accidents have usually been caused by some mechanical agency such as a frictional blow and, to the author's knowledge, none have been attributed to electrical discharges. This suggests that the method of test using condenser discharges between metal/metal electrodes in a circuit of low resistance does not reproduce the type of discharge taking place in practice. Discharges from the operator (the usual source of energy) will most likely take place from a finger, i.e. in a circuit containing at least one non-metallic electrode, and a resistance of about 100,000 ohms depending mainly on the condition of the skin. These two requirements are embodied in the present method of testing. The rubber base electrode consists of steel covered by a piece of conducting rubber fixed to it by conducting adhesive. This simple alteration changes one of the electrodes to a non-metallic

* lead 2:4 dinitroresorcinate

one and automatically introduces a resistance of approximately 100,000 ohms for a point contact. When tests are carried out with this arrangement, the low energy region of ignition of lead azide is eliminated, i.e. lead styphnate is now more sensitive than lead azide as is found in practice.

(d) With metal/metal electrodes and zero added resistance the minimum ignition energy is independent of capacity over quite a wide range. With lead styphnate, for example, the minimum ignition energy remains constant for the range 100 to 30,000 mmfd. Going below 100 mmfd, this energy decreases with decreasing capacity until a capacity of 55 mmfd is reached. Further reduction in capacity causes an increase in the minimum ignition energy. Above 30,000 mmfd, the energy increases, reference (e).

(e) When metal/rubber electrodes are used, the minimum ignition energy is no longer independent of capacity and varies over the whole range. There is also a minimum capacity for ignition, below which ignition will not take place, no matter what potential is placed on it. The resistance of the rubber causes the spark discharge to be split into several parts depending on the capacity and initial voltage. Consequently the individual discharges may or may not have sufficient energy in them to effect ignition. This becomes more important the smaller the capacity, and as a result a limit of capacity emerges, i.e. no matter what potential is placed on that capacity, the individual discharges will have insufficient energy to ignite the explosive, reference (c). With lead styphnate the minimum capacity is very small, with lead azide of high purity it is several hundred mmfd, and for dextrinated lead azide it is more than 1,000 mmfd.

4. The estimation of static hazard requires a somewhat long series of tests since the variation of minimum ignition energy has to be determined over a wide range of capacities. The results are best shown graphically. However, on the basis of present knowledge of sensitivity over the range of capacity up to 5,000 mmfd, it is possible to derive a fair estimate of hazard merely by determining the minimum energy at say 500 mmfd.

references (d) and (e). (This applies only to single compounds in a fairly pure state and not to mixtures.)

5. The following is a list of primary explosives in decreasing order of sensitivity, references (d) and (e): normal lead styphnate, tetracene, lead 2,4 D.N.R., lead azide of 97 percent purity, silver azide, mercury fulminate, and dextrinated lead azide.

Variance of NOL and E.R.D.E. Results

6. There was not complete agreement between the results obtained at NOL and E.R.D.E. for the sensitivities of basic lead styphnate and normal lead styphnate. For example, reference (g) showed that U. S. basic lead styphnate was apparently less sensitive than normal lead styphnate and the British forms of basic lead styphnate designated RD 1346 and RD 1349. This result seemed unlikely on the basis of U. K. experience, reference (e). Reference (h) gave the maximum energy for non-ignition of subsieve and milled normal lead styphnate as 13,250 and 70 ergs, respectively. Such large differences have not been encountered in the U. K. during the course of routine testing of a variety of samples of normal lead styphnate and the figures for maximum energy for non-ignition have always been in the region of the smaller figure, reference (e). Reference (g) indicated an energy of <62 ergs for the milled material (two ignitions were obtained in 10 trials with a capacity of 50 m[illegible]d charged to [illegible]0 volts).

New Investigations

7. In order to investigate these apparent anomalies it was decided to test the explosives under comparable sets of conditions. Also a need had developed for information in certain other areas. The following is a list of the additional explosives which were studied. They were either new to the author or they had not been tested by the metal/rubber method:

- DDNP
- Metal oxidant compositions based on zirconium, titanium, boron, or aluminum
- U. S. Tetracene
- KHND (Potassium Hexanitrodiphenylamine)
- DATB (Diaminotrinitrobenzene)

The last two explosives necessitated, for comparison purposes, trials on RDX, TNT, and tetryl.

8. In order to obtain comparable conditions, it was decided to build a replica of the U. K. apparatus rather than modify the existing NOL apparatus. The choice was made for the following reasons:

(a) An identical facility would be available at the two laboratories for comparison purposes.

(b) The NOL apparatus was used to test loaded components besides loose explosive and was best left untouched.

The construction details of the NOL version of the E.R.D.E. apparatus are given in paragraphs 9 through 19 below.

NOL VERSION OF THE E.R.D.E. APPARATUS

Electrode Assembly

9. A copy of the electrode assembly as described in reference (a) was made from linen phenolic sheet 1/4-inch thick, Figure 1. The movable arm was insulated from the remainder of the assembly by a Teflon sleeve, Figure 1, point A. The top half of the apparatus was insulated from the base by two sheets of polystyrene, Figure 1, point B.

10. Roye, Extra Loud phonograph needles were used for the upper needle electrode. Plumb-bob electrodes were made as described in reference (c). The base electrodes were solid cylinders of hardened steel 3/4-inch diameter, 3/4-inch high (i.e. similar to 3/4-inch rollers in a roller bearing). Discs of conducting rubber 1/2-inch diameter, 13/32-inch thick were cut from the stock held at E.R.D.E., Waltham Abbey, since there was no rubber available at NOL in the required range of resistance. These were fixed to the steel base electrodes by means of a conducting adhesive made from graphite powder mixed with the minimum of Goodyear Pliobond. The resultant resistance with a point contact was somewhat lower than 100,000 ohms, but as the resistance increases with use they were acceptable.

Condensers

11. Plasticon Glassmike condensers rated at 10 KV DC with capacities as indicated in Table 1 were used. These were fixed at point C, Figure 1, when required.

Table 1

List of 10 KVDC Capacitors

Nominal Capacity	No.	Actual Capacity
500 mmfd	3	470, 500, 500 mmfd
1,000 "	2	1,010, 1,050 "
2,000 "	1	1,850 "
5,000 "	1	4,050 "
0.03 mfd	2	0.0355, 0.0295 mfd
0.1 "	2	0.0939, 0.0957

For 245 mmfd, the 470-and 500-mmfd condensers were connected in series. For 167 mmfd, the 470, 500, and 500-mmfd condensers were connected in series. (This combination was used only on two occasions.) For 0.2 mfd, the two 0.1-mfd capacitors were connected in parallel. One larger capacitor, rated at 2 KVDC was also used. This had a nominal capacity of 0.25 mfd (actual 0.257 mfd).

12. For capacities below 100 mmfd, lengths of coaxial cable (Type RG8) were mounted on a wooden strip, the central wire terminal being suitably insulated from the board, with the sheath grounded, see Figure 2. For a capacity of 80 mmfd three lengths of 10-1/2 inches each were connected in parallel. For capacities of 25 mmfd, 10 mmfd and 5 mmfd, single lengths of 10, 4, and 2 inches, respectively were used. These coaxial cable capacitors could not be charged above 4 KV.

13. It was realized that the electrode assembly and leads probably had a capacity of several mmfd. When capacities of 25 mmfd or more were attached to the apparatus the contribution from the assembly and leads could be ignored. For capacities of 10 and 5 mmfd, the additional capacity makes a considerable contribution to the total capacity. However, no attempt was made to measure the capacity of the electrode assembly and leads, since the overall picture and conclusions would not be altered.

High-Voltage Unit

14. This was a conventional unit capable of giving potentials up to 10 KV DC with either combination, i. e. positive high potential with negative ground; or negative high potential with positive ground. A Sensitive Research Instrument Corporation voltmeter, ranges 0-30 KV, 0-7.5 KV and 0-15 KV, was used to determine the voltage output.

Technique for Checking Low-Voltage Discharges.

15. For potentials of less than 350 volts the discharge could occur without visual indication. It was therefore necessary to check whether or not a discharge actually took place when the upper electrode was brought down to the base electrode. This was accomplished by sharing any residual charge on that capacitor with another of suitable size so that the potential on the two of them could be measured on a 120-volt electrostatic voltmeter made by the Sensitive Research Instrument Corporation. (This instrument could not be used very satisfactorily with the capacitors of 5, 10, and 25 mmfd when charged to potentials under 500 volts. Its own capacity was much larger than 25 mmfd, and thus the potential was reduced to too low a figure for the instrument to register. It would have been preferable in these cases to use a Lindemann electrometer as this has a much smaller internal capacity.)

Firing Chamber and Sample Preparation Chamber.

16. The firing chamber and sample preparation chamber, Figure 3, were constructed from sheets of 1/4-inch Plexiglas and aluminum angle strips. The top, front, and sides were covered with sheets of 1/8-inch Homalite, a transparent plastic of reduced electrical resistance. Whenever this sheeting is rubbed, the voltage built up on it is much less than would be built up on Plexiglas and thus the chance of inducing dangerous charges on the operator due to inadvertent rubbing of the chamber was much reduced.

17. Sample preparation was carried out in the right-hand compartment. A grounded metal sheet was placed on the floor to ground the operator while he was handling the explosive.

18. The electrode assembly was placed in the left-hand compartment and the operator stood or sat on a stool, insulated from the floor by a rubber sheet. A microswitch was fitted to the door of the firing chamber so that the DC power supply was switched off whenever it was necessary to adjust anything inside the chamber, e.g. changing the needle or to clean up after a set of trials. All movements of the explosive samples were carried out from the outside, using the special handling tool, Figure 4. The latter was grounded whenever it was in use since the edges of the aperture through which it was placed were permanently grounded.

19. The chamber was connected to an existing exhaust-fan system by 5-inch ducting.

20. The laboratory space in which the tests were carried out could be dehumidified and experiments were not usually carried out in an atmosphere of greater than 45 percent relative humidity.

METHOD OF TESTING

Firing Procedure

21. Samples were prepared by placing about 5 to 15 mg. of explosive (depending on their bulk density) on the steel or rubber base electrodes, Figure 4. A filling plate technique, such as described in reference (c), is a precise way of obtaining a uniform charge; however, a spatula method was used in these experiments because most of the powders tested had poor flow properties, making the plate method unfeasible.

22. A condenser of appropriate capacity for the explosive under test was fixed to the electrode assembly as shown in Figure 1. The output of the high voltage unit was set to the potential to which the condenser was to be charged.

23. A sample was passed through the aperture between the firing chamber and the sample preparation chamber and placed on the platform of the electrode assembly. The condenser was then charged by rotating the handle, see Figures 1 and 3, so that the solid brass contact directly above the phonograph needle touched the source of high potential. When the voltmeter regained its original reading, the capacitor had been charged to the chosen potential. By rotating the handle in the opposite direction, the charged capacitor was then isolated from the charging circuit, and on further rotation the upper electrode was brought down towards the base electrode upon which the explosive lay. When the two electrodes were sufficiently near, a discharge took place.

24. Usually three trials were carried out on each sample of explosive on the base electrode. A fresh portion of explosive was exposed to the discharge by moving the electrode about on the platform. After the series of one, two, or three trials, depending upon whether or not the explosive fired, the base electrode was moved to a flat space to the right of the electrode assembly, Figure 3. A fresh sample was brought through the aperture from the preparation side. When the complete set of trials (normally 50) at one energy condition had been carried out, the

unconsumed explosive was disposed of by standard procedures, and the base electrodes were cleaned with emery paper, if necessary.

Test Plan and Criteria

25. Normally the sensitivity measurements were made using the "Frankford Run-down" type of test plan. Fifty trials were carried out at each test level. Each successive level was chosen in an effort to find a level at which there would not be initiations. It was observed that in most cases the distribution was non-normal. For instance, there are capacitor sizes, in the case of steel/rubber electrodes, where a certain percent of the trials lead to initiations but where voltage levels cannot be found at which 100 percent initiations can be observed . . . a sort of inherent dud rate. Since this whole program has been oriented toward developing information relevant to handling problems, it is desirable to get an estimate of the threshhold of sensitivity. Therefore, except as noted, the reported sensitivity level is the highest level at which no explosions were observed in fifty trials.

26 The degree of consumption of loose explosive varied from the emission of a small amount of smoke to a complete burning or detonation. The type of ignition was noted in all cases and it is usual to base the sensitivity on the evidence of the mildest ignition for safety purposes, since with a larger mass of material than is used in the test, a detonation or mass explosion may ensue.

Calculation of Energy

27. In this report, energies are quoted as stored energy (calculated from $1/2\ CV^2$) whether for the steel/steel or the steel/rubber electrodes. This is approximately the energy available in the discharge in the first case, (the efficiency of transfer of energy depending mainly upon the quality of the condenser). In the second case, however, the energy available in the spark gap is approximately 10 percent of $1/2\ CV^2$, the remainder being dissipated in the rubber, reference (b).

RESULTS

<u>Comparison of Results from the NOL-Built Apparatus with Those Found in the U. K.</u>

28. Ideally, comparison tests should have been carried out with the same samples of explosive as were used in the U. K. This was not possible, so explosives were chosen which were likely to behave similarly to two typical British primary explosives. These were U. S. normal lead Styphnate (used as received) to be compared with British normal lead styphnate RD 1303, and U. S. polyvinyl alcohol lead azide (used as received) to be compared with British Service lead azide.

29. The sensitivities of these two U. S. explosives were determined with the steel/rubber electrodes (rubber, positive ground; steel needle, negative high potential). Figure 5 compares the results obtained in the U. K. references (d) and (e) for normal lead styphnate RD 1303 and service lead azide with those obtained for U. S. normal lead styphnate and PVA lead azide on the new apparatus. In both cases the results compare very favorably, although for lead styphnate there is a difference for capacities below 50 mmfd. However, this is not felt to be a significant difference.

30. Tests with steel/steel electrodes showed that the minimum energy for U. S. lead styphnate was 125 ergs, which agrees with the values quoted in references (b) and (c). PVA lead azide was not examined under these conditions.

31. Thus, substantially identical results were obtained on the two pieces of apparatus.

<u>Effect of Polarity of the Conducting Rubber/Steel Electrodes</u>

32. Previous experiments had shown that with steel/steel electrodes at low energies, there was very little difference whether the needle electrode was positive or negative with respect to the base electrode, reference (c). There was a possibility, however, that this would not be the case when the base electrode was covered with conducting rubber.

33. Figure 6 shows how the spark sensitivities of normal lead styphnate and PVA lead azide changed when the polarity of the steel needle was changed from negative to positive. With lead styphnate the ignition energy values were halved throughout the range of capacity 25 mmfd. to 5,000 mmfd. However, this was not a large effect, and both sets of results fall within the area normally associated with the sensitivity of lead styphnate, reference (e). For milled normal lead styphnate, the difference was smaller, see next section.

34. For PVA lead azide the results with needle positive were again lower than those with the needle negative, and, since this compound has an appreciably higher minimum capacity for ignition than does lead styphnate, there was the additional effect of lowering this variable, i.e. from a value of about 400 mmfd. to one of about 200 mmfd.

35. It is possible that a capacity effect exists with lead styphnate, but, since the minimum capacities in this case are so small, any difference is not likely to be significant.

36. These tests show that there appears to be a small, but significant, effect with explosives such as lead azide since the minimum capacity for ignition is lowered. The effect with lead styphnate is not significant.

37. The reason for the difference with lead azide is not clear at present. It may be connected with the non-uniform distribution of energy in the gap, reference (c). It would be desirable to standardize the polarity, but this may have to await further work on the mechanism of ignition.

<u>Effect of Particle Size on Sensitivity</u>

38. Figure 7 shows the sensitivities of normal lead styphnate, unmilled as received, and milled, using steel/steel electrodes, with capacities of 245 mmfd and 500 mmfd. The minimum ignition energy (125 ergs) was the same for both particle sizes, but the percentage of ignitions rose more sharply in the case of the milled material as the energy was increased. Below 200 mmfd the minimum ignition energy for the milled styphnate dropped to 90 ergs at 80 mmfd, and 45 ergs at 25 mmfd. The NOL figure of <62 ergs for 50 mmfd fits fairly well, reference (g). The large value, 13,250 ergs, given for unmilled normal lead styphnate in reference (h) is difficult to understand.

39. Figure 8 shows the sensitivities of the unmilied and milled lead styphnate using steel/rubber electrodes, the steel needle being at positive or negative high potential. The curves for the unmilled styphnate are the same as those given previously in Figure 6. For capacities greater than 200 mmfd, there was little difference between the sensitivities of the milled material (needle positive or negative) and the unmilled material (needle positive). Below this capacity the difference became larger, and for capacities less than 80 mmfd, the energy required to ignite the unmilled styphnate increased; whereas, in the case of the milled material, it was still decreasing.

40. Lead azide with steel/steel electrodes gave decreasing minimum energies as the particle size was reduced, e.g. British Service lead azide gives 20 ergs, colloidal lead azide about 2 ergs, and for an azide intermediate in size, about 5 to 10 ergs, reference (c). However, this is felt to be partly a geometrical factor, since the smaller crystals will be nearer to the short arc discharge than the larger crystals. The needle pushes the larger crystals further away from the spot at which the discharge takes place.

<u>Sensitivity of Basic Lead Styphnate</u>

41. Using steel/steel electrodes (the upper one being a needle) and a capacity of 500 mmfd, partial ignitions of basic lead styphnate were obtained above 2,000 ergs. Below this figure only small amounts of the styphnate were ignited, as indicated by black burn marks on the rollers. There was no visible result for energies below 600 ergs. However, when a plumb-bob electrode was substituted for the needle electrode, partial ignitions were obtained down to 500 ergs. At this point it became difficult to discharge the condenser with this shape of electrode. By changing the capacity to 80 mmfd, so that larger voltages could be employed, partial ignitions were obtained down to 135 ergs, when it again became impossible to discharge the condenser.

42. Reference (g) showed that complete ignitions were obtained by charging a 50 mmfd capacity to voltages above 4,000 volts (i.e. above 4,000 ergs). With the particular electrode arrangement, it would not be easy to detect partial ignitions, i.e. if little smoke or light was emitted. It was observed that in attempting to destroy those samples of basic lead styphnate which had not ignited, energies far in excess of that for the predicted 100 percent ignition point were needed. This is likely if some of the styphnate had ignited in the trial, leaving a desensitized residue due to contamination with lead or carbon decomposition products. A further difference in the results is accounted for by the substitution of a plumb-bob electrode for a needle electrode.

43. With the steel/rubber electrodes (upper electrode being plumb-bob), it was more usual to obtain complete ignitions rather than partial ones. Though only a few trials were carried out, nearly all resulted in ignition at voltages which were capable of giving a discharge. Thus, a limit based on these experiments can be given, which, in the case of steel/rubber electrodes, is considerably more than the minimum ignition energy. Figure 9 shows the upper values for capacities of 25, 80, and 245 mmfd.

44. Before discussing these results, it will be necessary to compare results for the two forms of beta basic lead styphnate, RD 1346 and RD 1349, as obtained previously by NOL, reference (g), and by the author in the U. K., reference (e). Reference (g) indicated a value of <62 ergs for RD 1346, and approximately 100 ergs for RD 1349 (obtained by extrapolation) for the minimum energy using a capacity of 50 mmfd. These two figures agree fairly well with those obtained in the U. K., reference (d) Most RD 1346 samples gave values in the region of 75 ergs. The early samples of RD 1349 tested in the U. K. had a sensitivity less than normal lead styphnate as judged by results using steel/steel electrodes. Minimum energies of 200 to 500 ergs were obtained with the majority of the ignitions being partial ones. The indicated decreased sensitivity of RD 1349 as compared to other forms of lead styphnate was based on these results. However, as the method of preparation was improved so that RD 1349 could be made on a larger scale, the samples tended to be more sensitive, with energies of 150 to 200 ergs. When a representative sample of a large-scale preparation was tested, using the steel/rubber electrodes, similar results to those for normal lead styphnate were obtained, including a preponderance of complete ignitions. In fact there was little difference between normal lead styphnates RD 1302 and 1303, and beta basic lead styphnates RD 1346 and 1349, reference (e). The yellow form of alpha basic lead styphnate was more sensitive.

45. Since the figures given for the ignition limit for U. S. Standard basic lead styphnate (the alpha polymorph, reference (i)) using steel/rubber electrodes are likely to be at least 3 to 4 times too large, it can be concluded, taking all the results into consideration, that its sensitivity is about the same as normal alpha lead styphnate and the two beta basic lead styphnates.

Sensitivity of DDNP and Tetracene

46. The results for milled DDNP using steel/rubber electrodes are shown in Figure 10 along with comparative U. K. values for dextrinated lead azide, silver azide, lead 2:4 dinitroresorcinate and tetracene. The DDNP sample was rather old, and thus there is some doubt as to its purity. However, all ignitions were complete. Tests were not carried out on steel/steel electrodes.

47. Only a few trials were carried out on tetracene, but the minimum ignition energies for capacities of 500 and 1950 mmfd are greater than 10,000 ergs. The figures obtained for British tetracene I.C.I. at E.R.D.E. were 960 ergs at 120 mmfd, 2,300 at 500 mmfd, and 3,000 ergs at 1,000 mmfd, reference (f). This is form , which is usually obtained from commercial sources. Form B, which has been found to be present in some commercial samples, gives similar figures except at the smaller capacity end. The corresponding values are 4,800, 2,500, and 4,000 ergs reference (e).

48. Lattice plane spacings from an x-ray powder photograph of the U. S. tetracene were compared with those obtained from forms A and B. They suggested that the U. S. tetracene was form A, but the evidence was by no means conclusive.

49. The sample of U. S. tetracene was several years old and this may account for the decreased sensitivity as compared with the E.R.D.E. sample.

Sensitivity of Metal Oxidant Mixtures

50. The sensitivities of several metal/oxidant mixtures based on zirconium and boron have been investigated. Reference (a) indicated that the mix for the primer used in the Mk 10 Mod 0 Delay Element (based on zirconium) was sensitive. Reference (g) states that a layer of zirconium metal powder can be ignited with energies of less than 10 ergs.

51. The composition of the various zirconium mixtures tested for spark sensitivity in this series of tests is given in Table 2. In addition to this, a boron mixture (a Picatinny Arsenal delay composition) of boron/barium chromate (10/90) was also checked. Table 3 gives their minimum ignition energies using steel-steel electrodes. Figure 11 shows the steel/rubber results (needle positive).

52. Zirconium metal powder, the mix for the primer used in the Mk 10 Mod 0 Delay Element (hereinafter called "lead dioxide mix"), zirconium rich mixtures known as A1A (Universal Match and NAD versions) are the most sensitive and should be handled similarly to lead styphnate, i. e. with full antistatic precautions. The stoichiometric mixture F43B and the boron/barium chromate composition were somewhat less sensitive; they approached the sensitivity of PVA lead azide but they were capable of being ignited by smaller capacities. The sensitivity of the Army Chemical Center ZZ-50 mixture, which contains titanium was less than that of PVA lead azide.

53. It is of interest to look more closely into the sensitivities of mixtures containing zirconium. As noted above the steel/steel and steel/rubber results show that the order of decreasing sensitivity was as follows: Zirconium, A1A, F43B, ZZ-50; i. e. in the order of decreasing zirconium content.

Table 2

Composition of Zirconium Mixtures
Parts by weight

Composition	Zr	Fe_2O_3	Diatomaceous Earth [illegible]	PbO_2	Tetrazene	Ti
A1A	65	[illegible]	[illegible]	--	--	--
F43B	41	[illegible]	[illegible]	--	--	--
ZZ-50	17.5	[illegible]	--	--	--	[illegible]*
Lead dioxide mix	25.2	--	--	[illegible]	5.0	

* Plus binder of cellulose nitrate/camphor, 1.5 parts per 100 parts of metal plus oxide.

Table 3

Minimum Energies for Metal Powders and Metal/Oxidant Mixtures, Using Steel/Steel Electrodes

Composition	Minimum Energy (ergs)
AlA (Universal Match Corp.)	~12
AlA (NOL)	28
F33B	100
B2-50	4,500
Lead Dioxide Mix	47
Zirconium (Aerial Products Corp.) for mix above	15
$B/BaCrO_4$	550
Titanium, as used in B2-50	~ 50,000

This suggests that the first step in the ignition process is the ignition of zirconium metal in the oxygen of the air. This is followed by reaction with ferric oxide which needs a larger energy of initiation. In the case of the B2-50 mixture, where a large proportion of the zirconium is replaced by the less sensitive titanium, a much larger energy was required. Titanium powder of the size distribution for the B2-50 mixture gave the following energies for ignition: steel/steel, approximately 50,000 ergs; steel/rubber for a capacity of 0.0355 mfd, approximately 600,000 ergs. In the case of the lead dioxide mix which had about the same sensitivity as zirconium itself, the second stage reaction involving lead dioxide must require much less energy than the corresponding one with ferric oxide, and probably is similar to that required for ignition of zirconium in air. This seems plausible since lead dioxide is a much more reactive oxidant than ferric oxide.

54. As expected, there was a considerable difference in the rate of combustion of zirconium metal and the lead dioxide mix. In fact, combustion of the former and the ferric oxide mixtures on the rubber-topped electrodes was slow enough to char the rubber to such an extent that they could not be reused. This property makes some of these materials less hazardous to handle than would be indicated from a consideration of the ignition energies alone, although presumably a dust explosion of them could be very dangerous.

Sensitivity of Graphite-Coated Ammonium Perchlorate, Ammonium Perchlorate/Aluminum, and CJ Propellant

55. Tests were carried out on the following materials:

AP (passed 325 mesh, i.e. 44 microns)
AP coated with graphite (96/4)
AP/aluminum (75/25)
CJ propellant.

Samples of the first three were tested in the bulk state. Samples of the propellant were prepared by means of a cork borer from a slice of thickness varying from 0.02 to 0.04 inch. All samples were confined in collars. In the case of the steel/steel electrodes, the collars were fixed to the steel with adhesive. With the steel/rubber electrodes, the collar was placed in position on top of the rubber, no adhesive being used.

56. The plumb-bob type of upper electrode was used throughout. A condenser of 0.257-mfd capacity was used at voltages up to 2,000 volts. (Tests with steel/steel electrodes with a smaller capacity charged to higher voltages resulted in the samples of AP and AP mixtures being blown away before the passage of the spark, even though the collar was in place.) A few trials were carried out using a smaller capacity at higher voltages with the steel/rubber electrodes, and with the propellant on steel/steel electrodes.

57. Two groups of tests were carried out. The first was at room humidity (approximately 30 percent relative humidity) and with the sample as received. The second group was at somewhat lower humidities (e. g. 17 percent relative humidity obtained by placing dishes of phosphorous pentoxide in the chambers) and with the sample being used as soon as possible after having been dried overnight in a desiccator over phosphorous pentoxide.

58. AP/aluminum (75/25) was tested first at about 30 percent relative humidity. Partial ignitions were obtained above approximately 0.22 joule using steel/steel electrodes, see Table 4. At 13 to 20 percent relative humidity and overnight drying of the sample, partial ignitions were obtained above approximately 0.16 joule. These partial ignitions were judged mainly by an increase in the sound emitted over that of the spark discharge itself. Consumption of material gave no clue since most of it was blown out of the collar by the discharge anyway. With steel/rubber electrodes no ignitions were obtained. No ignitions were found

with AP, AP/graphite (96/4), or with the propellant as indicated in Table 5.

59. It is possible that a fine dispersion of the AP/fuel mixture in air could be ignited with smaller energies, although not necessarily so, since larger energies were needed for dust clouds than for the layers of magnesium and zirconium, reference (j). The potential difference between the two electrodes causes some of the AP/fuel sample to be blown away up from the base electrode and dispersed in the air within the collar when the discharge takes place. The test probably covers both conditions, i. e. an undisturbed layer and an aerial dispersion. Reference (j) gives a minimum energy value of 0.05 joule for the most hazardous sample of aluminum tested as a dust cloud. Reference (k) gives 0.047 joule for a dust cloud of aluminum when a resistance of 75,000 ohms is in series, the size of the aluminum being such that 90 percent passed a 200 U.S.S. sieve (76 microns).

60. Since the energies involved in the one case where partial ignitions were obtained, i. e. AP/Aluminum mixtures, were larger than the above figures, and much larger than those capable of igniting primary explosives, it is very unlikely that accidental ignition of AP mixtures by static discharge would occur.

Table 4

Ignition vs. Energy for AP/Aluminum(75/25)

Humidity	30	30	30	18	18	19	30	15	30
Base Electrode	S	S	S	S	S	S	R	R	R
Capacity, mfd.	0.257	0.257	0.257	0.257	0.257	0.257	0.257	0.257	0.0355
Potential, KV	1.8	1.5	1.4	1.3	0.2	1.1	2.0	2.0	7.0
Energy, Joules	0.42	0.29	0.25	0.22	0.19	0.15	0.51	0.51	0.87
Ignitions	5/5	1/5	1/10	3/3	2/5	0/5	0/21	0/15	0/5

S = steel
R = rubber

Table 5

Ignition vs. Energy for AP, AP/Graphite, and GR Propellant

Material	b	b	b	b	d	d	d	a
Humidity	30	17	30	15	30	30	30	30
Base Electrode	S	S	[illegible]	R	S	S	R	R
Capacity, mfd.	0.257	0.257	0.257	0.257	0.257	0.0355	0.0355	0.257
Potential, KV	2.0	2.0	2.0	2.0	2.0	9.0	9.0	2.0
Energy, Joules	0.51	0.51	0.51	0.51	0.51	1.44	1.44	0.51
Ignitions	0/13	0/3	0/21	0/15	0/5	0/5	0/5	0/15

S = steel
R = rubber
a = AP (<4 microns)
b = AP coated with graphite 96/4
d = GR propellant
Humidity = percent relative humidity

61. The sensitivities of RDX, tetryl, PETN, KHND, and DATB were tested in the bulk state. Of these only PETN and DATB gave complete or nearly complete ignitions, with energies of less than 1 joule. RDX and tetryl gave, at most, an indication of ignition by emitting some smoke. However, these required energies of 1 to 1.25 joules. KHND was inert up to 1.5 joules.

62. The detailed results are as follows, plumb-bob electrodes being used throughout.

(a) RDX, Tetryl, and KHND. The results are given in Table 6. The tests were conducted with and without collars. Since there was no marked difference in the result, this variation in the condition has not been included in the Table.

Table 6

Ignition vs. Energy for RDX, Tetryl, and KHND

Explosive	Electrode	Ignitions vs. Trials						
		Pot. (kV)	2	3	4	4.5	5	10
		E (Joules)	0.2	0.45	0.8	1.01	1.25	1.5
RDX	S		-	0/1	0/1		1*/4	-
	R		-	-	0/5	2*/5	2*/5	
Tetryl	S		0/2	0/2	-	-	0/5	-
	R		-	-	-	0/5	1*/5	-
KHND (Large)	S		-	-	-	-	0/3	-
	R		-	-	-	-	0/3	-
KHND (Small)	S		-	-	-	-	-	-
	R		-	-	-	-	0/3	0/2

* Denotes smoke emitted.

(b) PETN and DATB. The results are given in Table 7. Except in the case of PETN with steel/steel electrodes there was no confinement. With DATB there was no movement of the explosive under the influence of the electric field and very little movement as a result of passage of the electric discharge. Thus confinement was unnecessary. PETN either

gave an easily detectable amount of smoke or a nearly complete ignition. With DATB, four degrees of ignition were found:

(1) A very small amount of smoke, i.e. smoke that was just visible (denoted as V.S.).

(2) A small amount of smoke, i.e. smoke that was easily detectable, leaving most of the explosive untouched (S).

(3) A partial ignition, i.e. about half the material disappeared with an appreciable "crack" (P).

(4) An almost complete ignition, i.e. nearly all the material ignited with a correspondingly louder "crack" (C).

Table 7 shows that the results obtained with DATB and PETN were quite similar. With DATB appreciable reaction started occurring at energies in the order of 0.5 joule; with PETN appreciable reaction was occurring in the energy range of 0.6 to 1.25 joules. Although the testing is not complete, at the energies considered DATB has a comparable sensitivity to PETN and similar precautions should apply. KHND should not present any static hazard.

63. The results given in Tables 6 and 7 show that with Booster and high explosives it would be preferable to supplement the above tests with ones using more vigorous conditions of confinement and spark energy.

Table 7

Ignition vs. Energy for DATB and PETN

Explosive	Base Electrode	Capacity (mfd.)	Potential (KV)	Energy (Joules)	No. of Trials	Smoke VS	Smoke S	Ignition P	Ignition S
DATB	S	0.2	4.0	1.6	10	0	1	0	0
	S	0.2	5.0	2.5	10	0	2	0	0
	R	0.2	2.0	0.4	10	7	0	0	0
	R	0.2	3.0	0.9	10	0	0	0	0
	R	0.2	4.0	1.6	10	0	5	0	0
	R	0.2	5.0	2.5	10	0	5	0	5
	R	0.03	3.0	0.135	10	2	0	0	0
	R	0.03	4.0	0.24	10	3	0	0	0
	R	0.03	5.0	0.375	10	0	3	0	0
	R	0.03	6.0	0.54	10	0	7	0	0
	R	0.03	7.0	0.735	10	1	3	3	0
PETN	S *	0.03	2.4	0.085	5	0	0	0	0
	S *	0.03	2.6	0.099	4	0	0	0	1
	S *	0.03	2.7	0.103	1	0	0	0	1
	R	0.1	3.5	0.61	5	0	1	0	0
	R	0.1	4.0	0.80	5	0	2	0	0
	R	0.1	4.5	1.01	5	0	2	0	0
	R	0.1	5.0	1.25	6	0	3	0	0
	R	0.2	2.7	0.73	5	0	0	0	0
	R	0.2	3.0	0.9	5	0	1	0	0
	R	0.2	5.0	2.5	5	0	0	0	0

* with collar

CONCLUSIONS

64. The NOL-built replica of the E.R.D.E. apparatus appears to behave in an identical manner to the original, and thus can be used for comparison purposes. It is felt that this method of testing primary explosives, involving the use of a conducting rubber base electrode, provides a realistic measurement of electrostatic hazard. Oscillographic experiments, reference (e), showed that the discharge from a condenser in a circuit containing the rubber electrode was very similar to that obtained from a charged human being.

65. From the series of tests reported here, it can be concluded that

(a) The sensitivity of U. S. Standard alpha basic lead styphnate is about the same as normal lead styphnate and as the British forms of beta basic lead styphnate designated RD 1346 and RD 1349.

(b) There is no large variation in sensitivity of normal lead styphnate with particle size.

(c) The sensitivity of DDNP lies between that of lead 2, 4-dinitro resorcinate and lead azide (PVA type).

(d) The sensitivities of U. S. tetracene and U. K. tetracene are not identical. However, this may be due to the difference in age of the two samples.

(e) Small sensitivity differences are found depending upon whether the rubber base electrode is at positive or negative high potential.

(f) The sensitivities of various metal/oxidant mixtures based on zirconium, boron, and titanium fall in the range usually associated with primary explosives, and appropriate precautions should be taken in handling them.

(g) Of the mixtures based on ammonium perchlorate, only the AP/Aluminum (75/25) mixture could be ignited below 0.5 joule, and these were partial ignitions. Thus AP mixtures are unlikely to present an electrostatic hazard.

(h) Of the following booster and high explosives — RDX, tetryl, PETN, NHND, and DATB — only PETN and DATB gave complete or nearly complete ignitions with energies of less than 1 joule. RDX and tetryl emitted a little smoke above 1 joule. AHND was inert up to 1.5 joules. Thus under certain conditions it might be necessary to take precautions with PETN and DATB, but these are unlikely to be needed with any of the other explosives.

ACKNOWLEDGEMENTS

66. The author wishes to thank the members of the Initiation Research group of the Explosion Dynamics Division for their help in constructing the replica of the U. K. apparatus, and particularly Mr. Samuel Stackhouse for carrying out the trials described in this report. We would also like to thank Mr. H. L. Austin, NOL representative in the U. K., for his cooperation in forwarding the discs of conducting rubber from Dr. D. P. Scaife, E.R.D.E., Waltham Abbey, England; Mrs. Selma Ullman (US Division) for carrying out the x-ray powder photograph of tetracene; Mr. J.R.C. Duke (E.R.D.E.) for supplying details of his x-ray spacings on the two forms of tetracene, and Dr. J. R. Holden for discussions on the interpretation of the two sets.

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(c) Part II (of above report), 5/R/56. – [illegible]

(d) Part III (of above report), 22/R/56. – AD – [illegible]

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(g) V. J. Menichelli, A Comparative Study of the Static Spark Sensitivity of Several Forms of Basic Lead Styphnate and Priming Mixtures Containing Zirconium Metal and Zirconium Hydride, NAVORD Report [illegible], AD-117 340 August 1956.

(h) Ordnance Explosive Train Designers Handbook, NOLR 1111.

(i) F. Taylor, Jr., Primary Explosives Research VII, Free Flowing Basic Lead Styphnate, NAVORD Report 4442, [illegible] December 1956.

(j) [illegible], Industrial & Engineering Chemistry, 49, 752, 1957.

(k) A. R. Boyle and F. J. Llewellyn, Journal of the Society of Chemical Industry, 69, 173, 1950.

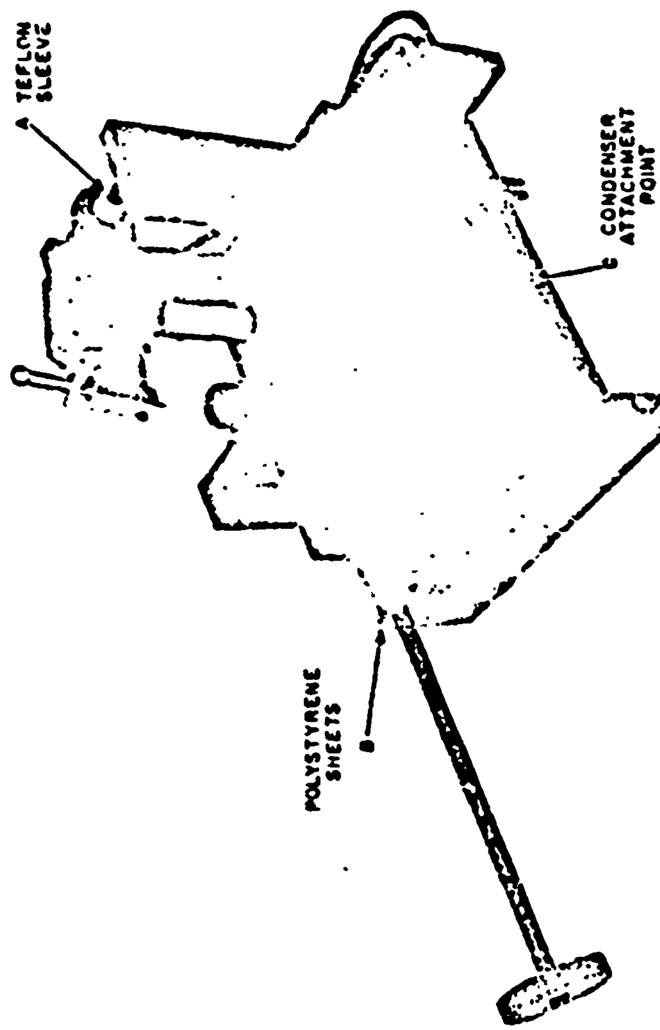

FIG 1 ELECTRODE ASSEMBLY

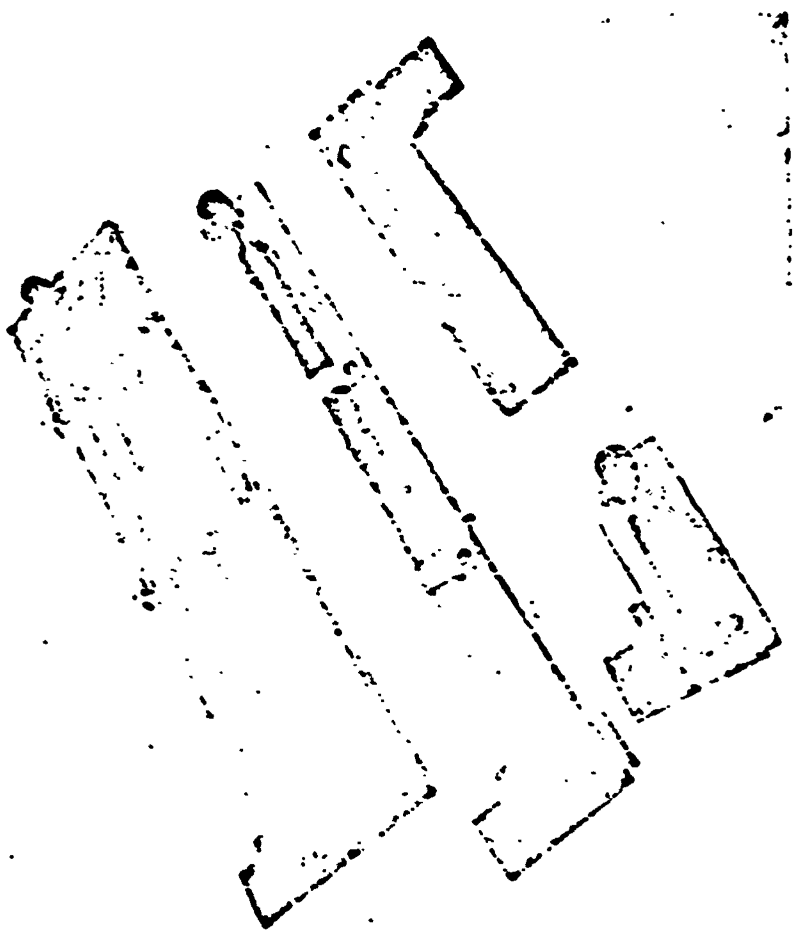

FIG 2. COAXIAL CABLE CAPACITORS

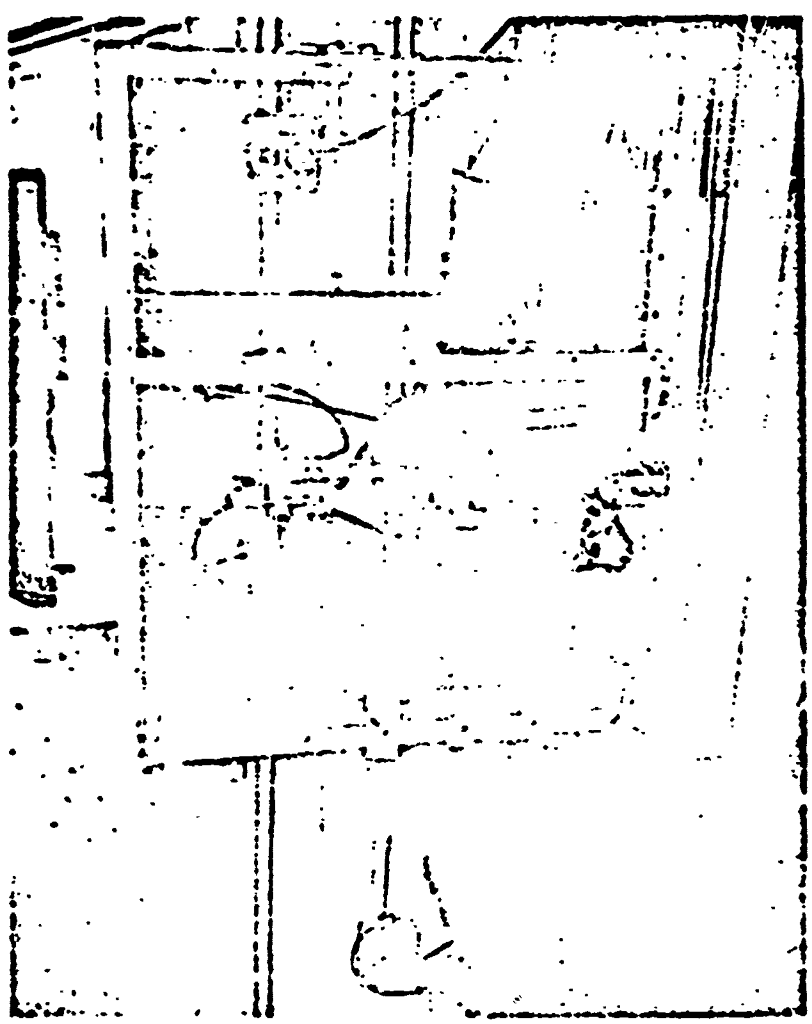

FIG 3. FIRING AND PREPARATION CHAMBER

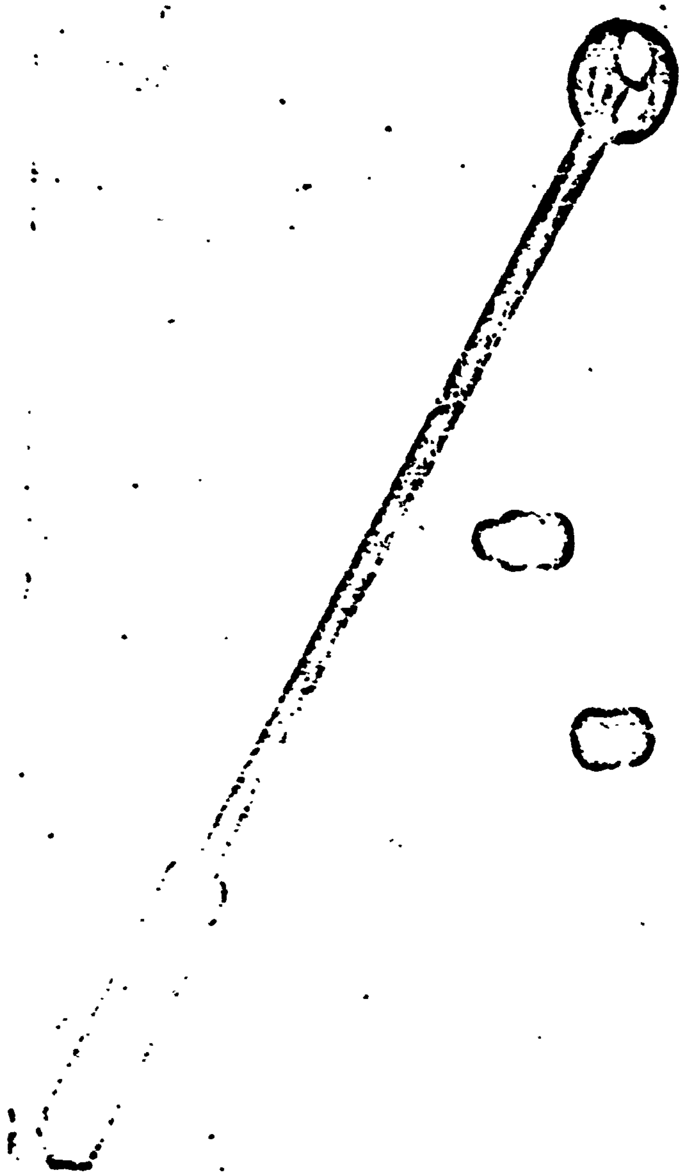

FIG 4. HANDLING TOOL AND BASE ELECTRODES

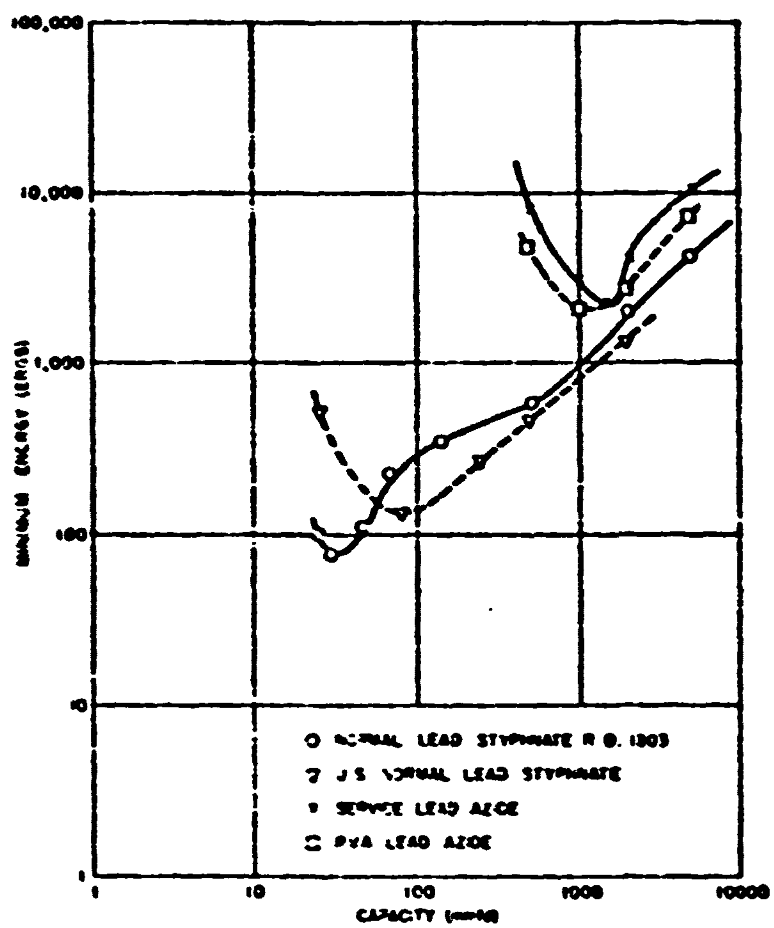

FIG 5 SENSITIVITY OF NORMAL LEAD STYPHNATE AND LEAD AZIDE MEASURED ON UK AND US APPARATUS

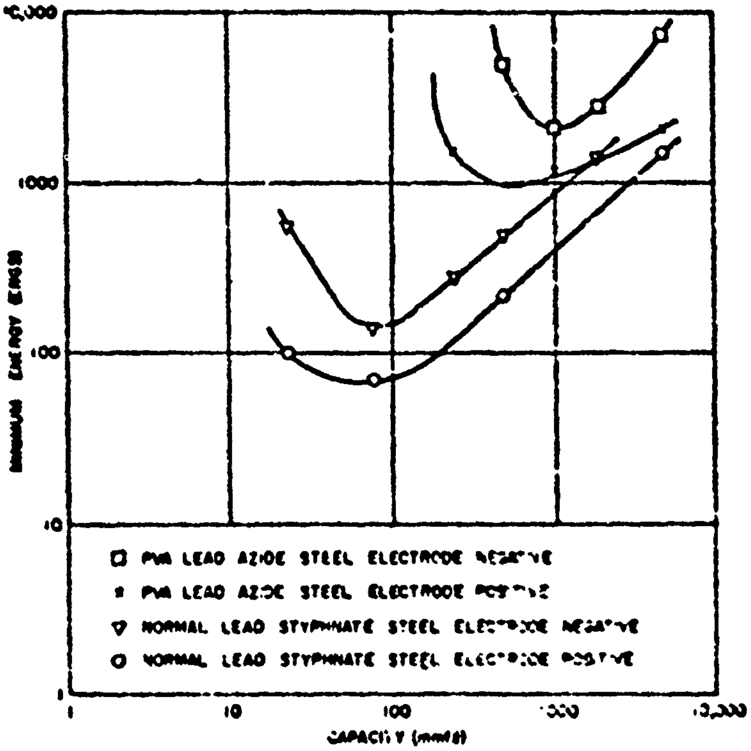

FIG 6. EFFECT OF POLARITY ON MINIMUM OF ENERGY
(STEEL/RUBBER ELECTRODES)

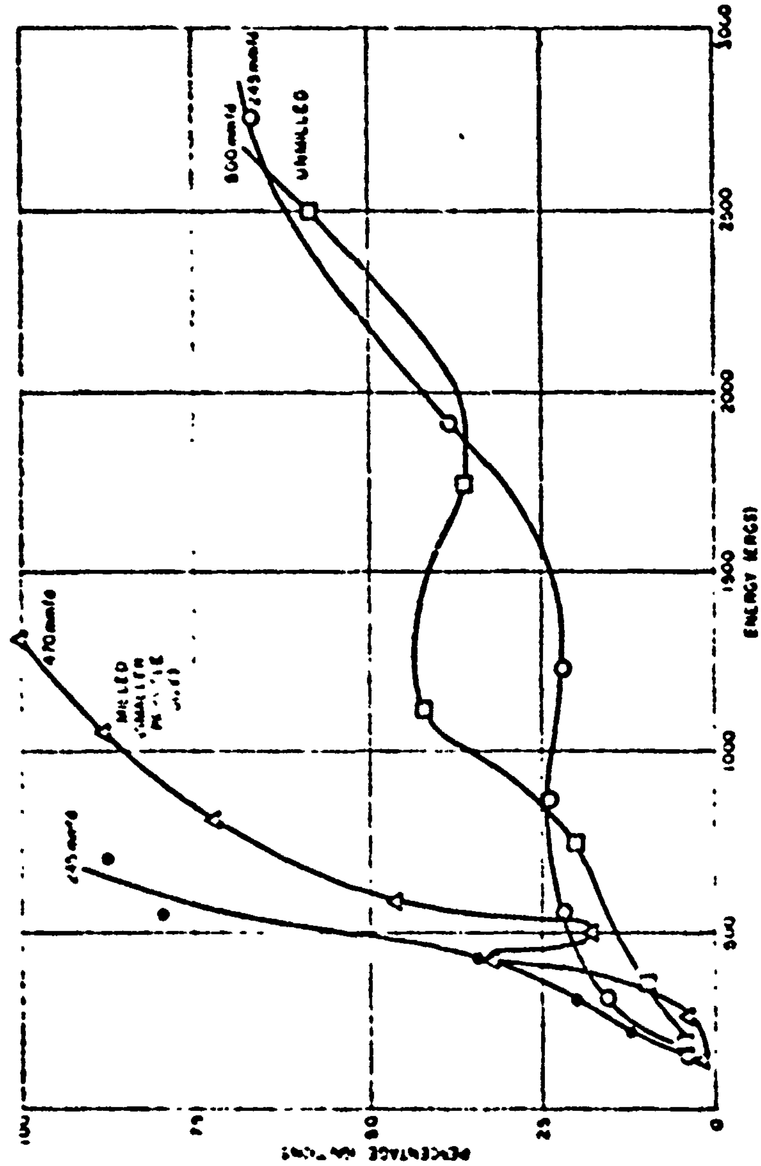

FIG. 7 SENSITIVITY OF MILLED AND UNMILLED NORMAL LEAD STYPHNATE STEEL/STEEL ELECTRODES

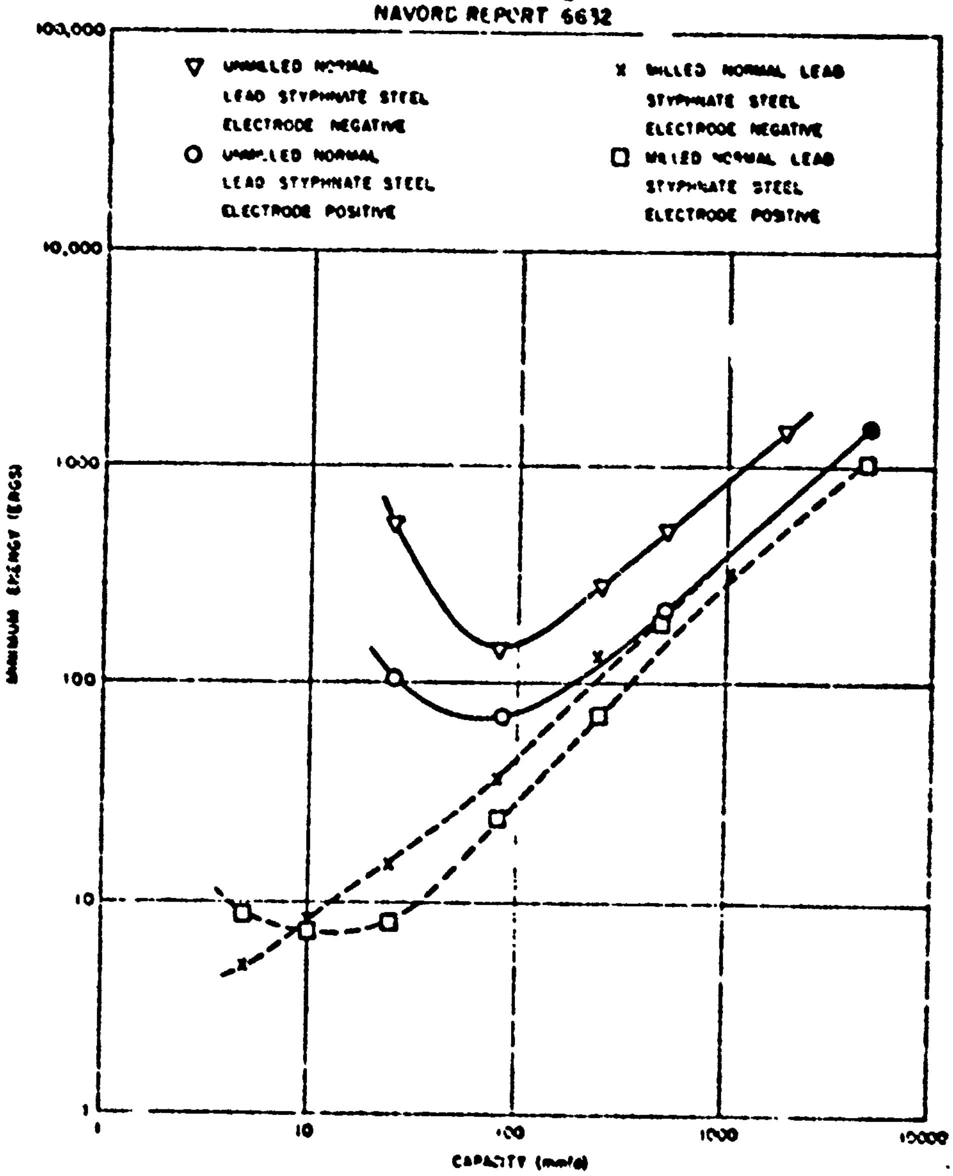

FIG. 8 SENSITIVITY OF MILLED AND UNMILLED NORMAL LEAD STYPHNATE (STEEL/RUBBER ELECTRODES)

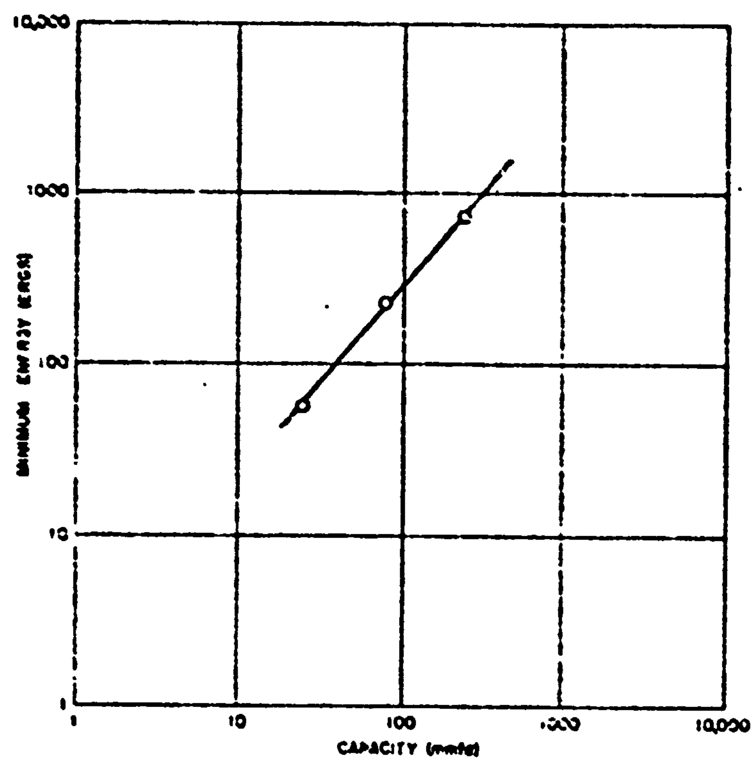

FIGURE 9 SENSITIVITY OF BASIC LEAD STYPHNATE
(STEEL/RUBBER ELECTRODES)

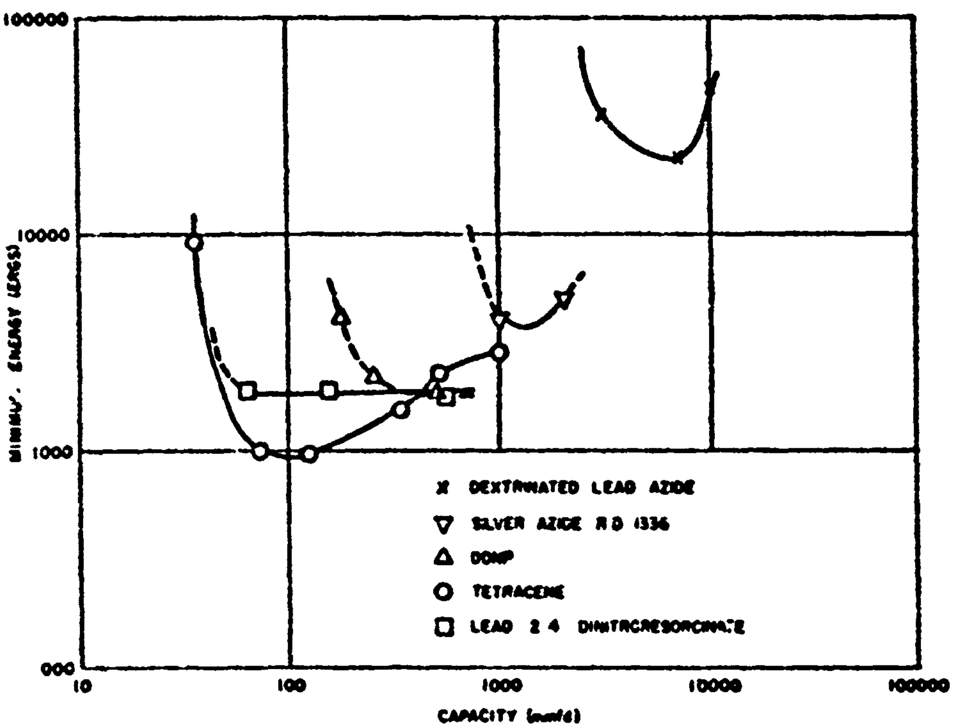

FIG. 10 SENSITIVITY OF DDNP COMPARED WITH UK VALUES FOR TETRACENE, DEXTRINATED LEAD AZIDE, SILVER AZIDE, RD 1336, AND LEAD 2:4 DINITRORESORCINATE

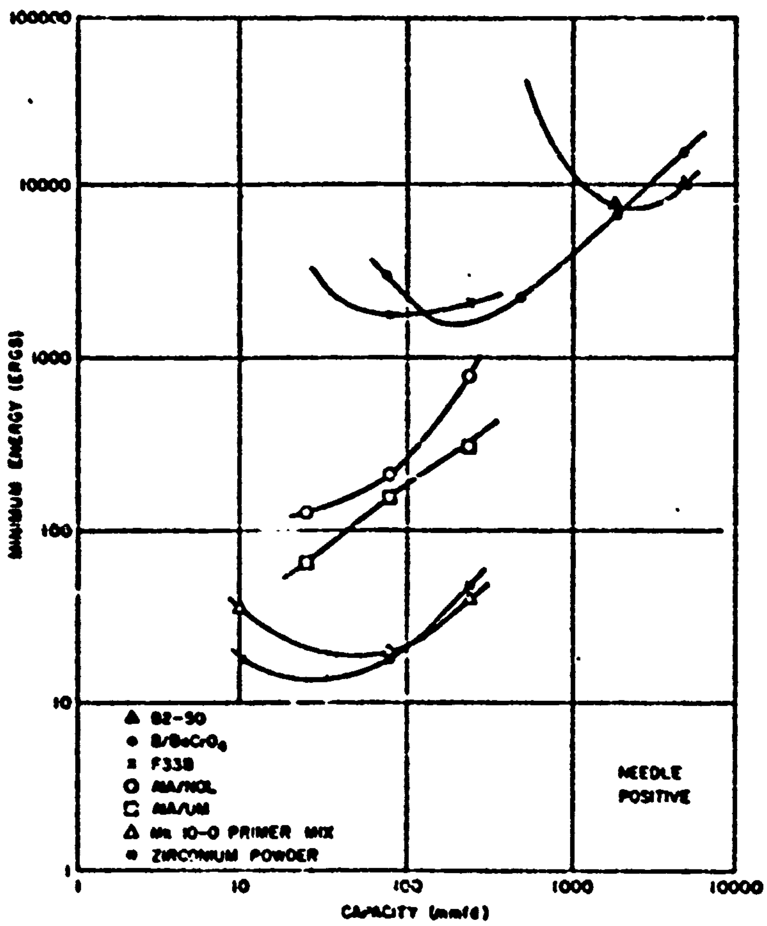

FIGURE 11. SENSITIVITY OF METAL-OXIDANT MIXTURES (STEEL/RUBBER ELECTRODES)